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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/US93/03551 <b>(22) International Filing Date:</b> 14 April 1993 (14.04.93)  <b>(30) Priority data:</b> 07/881,852                      12 May 1992 (12.05.92)                      US  <b>(71) Applicant:</b> MINNESOTA MINING AND MANUFACTURING COMPANY [US/US]; 3M Center, P.O. Box 33427, Saint Paul, MN 55133-3427 (US).  <b>(72) Inventor:</b> WILSON, Vernon, H. ; 3M Center, P.O. Box 33427, Saint Paul, MN 55133-3427 (US).  <b>(74) Agents:</b> WENDT, Jeffrey, L. et al.; Office of Intellectual Property Counsel, Minnesota Mining and Manufacturing Company, P.O. Box 33427, Saint Paul, MN 55133-3427 (US).		<b>(81) Designated States:</b> JP, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> FIRE PROTECTIVE FLEXIBLE COMPOSITE, SYSTEM INCLUDING SAME METHOD OF MAKING THE COMPOSITE, AND METHOD OF FIRE-PROOFING  <b>(57) Abstract</b>  Previously known fire resistant materials are not suitable for use in dynamic (moving or subject to movement) structures. Flexible composite materials of the present invention are suitable for use as fire barriers for either static or dynamic joints and are cost effective and easy to install. In one preferred embodiment the flexible composite includes: (a) a first layer material having first and second major surfaces, the first layer material including inorganic fibers and a binder in the form of a flexible mat; (b) a second layer material adhered to the first major surface of the first layer material, the second layer material being metal foil, the metal foil having a melting temperature of at least 350 °C; and (c) a third layer adhered to the second major surface of the first layer material, the third layer material including an intumescent fire retardant composite material.		

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**FIRE PROTECTIVE FLEXIBLE COMPOSITE, SYSTEM INCLUDING  
SAME, METHOD OF MAKING THE COMPOSITE,  
AND METHOD OF FIRE-PROOFING**

5

**Field of the Invention**

This invention pertains to flexible composite materials suitable for use in deterring the spread of fire, smoke, and fumes as may happen in a fire in a multi-floor building.

**Background Art**

10 Fire, smoke, and fumes in confined spaces, such as multi-floor buildings, can be extremely life threatening. Frequently, if fire originates in the space between a floor and ceiling of such a structure, the fire, and resultant smoke and fumes, will tend to spread to other open spaces in the building, especially to open spaces above the point of origin of the fire.

15 The reasons behind this spread of fire, smoke, and fumes to higher areas are varied. The areas between conduits, piping, and the like, and floors/ceilings through which they pass, are known as "through-penetrations". If not fire protected, through-penetrations offer areas of low resistance to fire, smoke, and fumes, and in essence serve as "chimneys". These areas may be  
20 filled with commercially available fire retardant and intumescent putties, caulks, wraps, or mats, known in the art as "firestops". Representative firestop products are disclosed in product brochure number 98-0701-3508-6 (published 1990) from Minnesota Mining and Manufacturing Company (3M). The 3M products are currently known under the trade designations "CP 25WB", "CP 25  
25 N/S", "CP 25S/L", and "Firedam" (caulks); "MPP-4S" and "MPS-2" (moldable putties); "FS-195" and "CS-195" (moldable strips); and "Interam" and "Interam E-5" (mats). These products are variously described in assignee's U.S. Pat. Nos. 3,916,057; 4,273,879; 4,305,992; 4,364,210; 4,433,732; and 4,467,577.

30 The above firestop products and others have been widely used for reducing or eliminating the chimney effect for through-penetrations and pass the

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rigorous American Society of Testing Materials (ASTM) fire endurance test (ASTM E-814) after intumescenting and charring. However, even if the fire is contained in the space between one floor and the next highest floor by a firestop, serious hazards remain. This is because many multi-level buildings

5 will have joints between exterior walls and floors constructed as illustrated in side elevation in FIG. 1. Shown is "vision" or "spandrel" (i.e., ornamental) glass 10, which may form the exterior of a building. (Alternatively, 10 may be concrete, marble, and the like.) Typically, an inorganic fibrous material 12 is installed for thermal insulation (referred to in the art as a "curtain wall"). The

10 inorganic fibrous material may be glass fiber, mineral wool, and the like. Thermal insulation 12 is fastened to a "mullion" 13 (term of art for the metal frame system for the exterior glass and thermal insulation) with screws or other means as shown at 16 and 18. Also shown is a concrete floor slab 20 which is typically supported by an I-beam 22. A "safing" material 14 is also typically

15 installed, which may be glass fiber, mineral wool, or other type of inorganic fibrous material insulation. One or more Z-clips 15 is typically provided for mechanically supporting safing 14.

It is important to note that an air space 24 is left in the construction illustrated in FIG. 1, between the mullion, thermal insulation, and the vision or

20 spandrel glass (typically about 2.5 cm gap). As heat is generated in the interior of the building in the vicinity of such a wall/floor joint, if the temperature is high enough, the binder in mineral wool insulation will oxidize, exposing the air space 24 to fire, smoke, and fumes, and allow the chimney effect discussed above. (Glass fiber insulation will begin to disintegrate at about 565°C,

25 causing similar problems.) Heat from the fire may then distort the mullion system, cause the concrete floor to deform, and may ultimately cause the vision or spandrel glass (or other exterior wall material) to shatter. Obviously, falling debris present a hazard to people outside of the building, such as fire control personnel and on-lookers, and fire hoses may be cut by falling glass shards and

30 other debris. Thus, it would be highly advantageous to keep the temperature of the thermal insulation as low as possible, for all of these reasons.

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As explained by Nicholas, J.D., in "Making Joint Systems Fire-Resistive", NFPA Journal, March/April (1991), pp.98-102, at 100:

5 The crucial difference between joints [such as those illustrated in FIG. 1] and through- penetrations is movement. Firestops are designed for static applications because the movement of penetrating items, such as pipes, is normally absorbed by bellows joints and directed away from the firestop. Thus, the firestop remains relatively static. However, joints do move, responding to expansion, contraction, shear, and rotational joint movements  
10 caused by thermal variations, seismicity, settlement, and wind sway. . . . If the fire barrier deteriorates, permanently deforms, or cannot cycle, it may not be able to maintain its fire rating.  
(Emphasis supplied)

15 Note that the terms "firestop" and "fire barrier" have different and precise meanings in the art, the former describing materials used in through-penetrations and other static joints, the latter used to denote materials used in movable (dynamic) joints.

There is thus a requirement for a flexible composite material which can  
20 be used in conjunction with conventional thermal insulation to form a system which provides not only adequate thermal insulation under static conditions, but which also provides the required fire barrier properties for dynamic joints such as illustrated in FIG. 1. The present invention is drawn toward meeting this need. Currently, as explained by Nicholas at page 100, there exists no fire  
25 endurance standards for fire barriers since standard tests have not been available.

U.S. Pat. No. 4,977,719 (LaRoche et al.) describes an expansion joint for interior or exterior use including a fire barrier comprised of a fire resistant inorganic refractory fabric sheet which supports resilient fire resistant inorganic  
30 refractory fibers. German patent application DE 3632648 (Figen et al.) describes a rain-proof and fire-resistant movable profile system for a freely movable connection of an available building wall, especially a wall of an older building, and a butt-jointing attachable exterior wall, consisting of three individual profiles which can be moved with respect to each other. Neither

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reference suggests the composites, systems, or methods of the present invention.

#### Summary of the Invention

It has now been determined that wall/floor joints such as those illustrated  
5 in FIG. 1, and other "dynamic" (subject to movement) joints, may be brought  
into compliance with an equivalent of ASTM E-119 through their entire cycle  
of expansion and contraction. Certain preferred embodiments of the novel  
flexible composite materials and systems of this invention provide this long felt  
need. These preferred embodiments will not only pass the rigors of a fire  
10 endurance test comparable to ASTM E-119, but will also pass the hose stream  
test of this standard. Construction joints (static joints between two floor slabs)  
would also benefit from these constructions; however, as movement is not a  
problem in this type of joint, other preferred flexible composite materials and  
system embodiments within the invention will be sufficient to pass the E-119  
15 test.

In its broadest embodiment, the invention comprises a flexible composite  
material suitable for use in static joints, the flexible composite characterized by  
having:

(a) a first layer of material having first and second major surfaces, the  
20 first layer material being inorganic fibers and a binder in the form of a flexible  
mat; and

(b) a layer of metal foil adhered to the first layer by an adhesive, the  
metal foil having a melting temperature of at least about 350°C. These flexible  
composite embodiments are suitable for use as a fire barrier in static conditions.

25 Preferably, when used as a fire barrier for dynamic joints, such as  
wall/floor joints experiencing thermal expansion/contraction cycling, the  
flexible composite material includes a third layer (c) adhered to the second  
major surface of the first layer material, the third layer material being a flexible  
intumescent fire retardant composite. This embodiment may also be used in  
30 static joints.



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In all embodiments of the invention the inorganic fibers of the first layer are selected from the group consisting of alumina-silicate fibers, mineral wool fibers, glass fibers, and refractory filaments such as zirconia-silica fibers and crystalline alumina whiskers. Mixtures of these inorganic fibers are also usable within the invention. The first layer may optionally include up to 65 weight percent (More preferably from 40 to 65 weight %) unexpanded vermiculite. Binders may be organic and/or inorganic.

An alternate embodiment of the flexible composite material of the invention, also suitable for use as a fire barrier for static joints, is characterized by having:

- (a) a first layer material having first and second major surfaces, the first layer material being inorganic fibers and a binder in the form of a flexible mat, as above described;
- (b) a second layer material adhered to the first major surface of the first layer material, the second layer material being a flexible intumescent fire retardant composite material as above described;
- (c) a third layer adhered to the second layer, the third layer being inorganic fibers which are the same or different than the first layer as above described; and
- (d) a fourth layer adhered to the third layer, the fourth layer being a flexible intumescent fire retardant composite material.

Another alternate embodiment of the flexible composite material of the invention suitable for use with static joints is characterized by having:

- (a) a first layer material having first and second major surfaces, the first layer material being inorganic fibers and a binder, formed as a flexible mat, as above described;
- (b) second and third layers adhered to the first and second major surfaces of the first layer, respectively, the second and third layers being inorganic fibers which are different from those of the first layer;
- (c) fourth and fifth layers adhered to the second and third layers, respectively, the

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fourth and fifth layers being a flexible intumescent fire retardant composite material.

Systems for thermally insulating and providing fire barrier properties for an exterior wall and/or an exterior wall/floor joint of a building are also  
5 presented. All systems of the invention are designed to be installed in buildings wherein the wall comprises mullion and exterior material, and wherein the floor comprises a material which is rigid at room temperatures but experiences deformation due to thermal expansion (especially at fire temperatures) and contraction, seismic activity, and the like.

10 The first preferred class of systems according to the invention may be described generally as "long" versions, since the fire barrier extends the entire length of the insulating component. This embodiment of the system is characterized by having:

(a) an insulating component positioned within the shape defined by the  
15 mullion upon attachment to the mullion and having interior and exterior facing surfaces, the insulating component being an inorganic material capable of providing thermal insulation for the building;

(b) a safing component positioned between an exterior butt end of the floor and the insulating component; and

20 (c) a fire barrier characterized by having a flexible composite material, the fire barrier having first and second portions, the first portion positioned adjacent and parallel to the insulating component, and the second portion positioned adjacent the safing component upper surface, the second portion having first and second ends, the first end attached to the top surface of the butt  
25 end of the floor and the second end attached to the first portion, and the first portion of the fire barrier attached to the mullion, wherein the second component has at least one curved portion which provides slack, thus allowing the fire barrier to effectively lengthen and shorten during relative movement of the wall and floor.

30 A particularly preferred system of this class of systems is the system wherein the first portion of the fire barrier has an upper and a lower terminus

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and is positioned adjacent and parallel to the interior facing surface of the insulating component. The lower terminus of the first portion is attached to the mullion in a fashion which completely covers the insulating component. The upper terminus of the first portion is attached to the second fire barrier portion.

- 5 The upper terminus extends between the insulating and safing components in this embodiment. The second portion most importantly includes an S-shaped curved portion or like accumulation which is positioned adjacent the upper surface of the safing; intumescent caulk is applied to seal the fire barrier to the insulation and to the safing, and the safing is supported by one or more Z-clips
- 10 or similar clips. This system is particularly preferable as a retrofit or original installation when the insulating component is glass fiber. These "long" versions also prolong the life of the exterior glass or other material, as well as the mullion.

A method of making the three layer version of the flexible composite

15 materials of the invention includes the steps of

- i) providing a laminate comprising a first layer, the first layer being inorganic fibers and a binder formed into a mat, the first layer having on one major surface thereof a second layer consisting essentially of metal foil adhered to the first layer by an adhesive;
- 20 ii) coating an intumescent precursor solution which includes a solvent and an intumescent fire retardant material onto the second major surface of the first layer to produce a wet coated composite material; and
- 25 iii) exposing the wet coated composite material to conditions sufficient to cure the intumescent fire retardant material, thereby forming a flexible third layer which includes the intumescent fire retardant material.

Preferably the solvent is water or an organic solvent, and the step of exposing the wet coated composite material to conditions sufficient to cure the

30 intumescent fire retardant material is by the application of heat.

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In one method of installing the system, insulating material is first positioned in the mullion. The flexible composite having three layers is then placed adjacent and parallel to the surface of the insulation facing the interior of the building, securing the lower end of the composite to the mullion, being sure  
5 that the flexible composite completely covers the lower terminus of the insulation. With the intumescent fire retardant side of the composite facing toward the interior of the building, the composite is positioned between the insulation and safing, after which the composite is accumulated in an "S" shape (or the like) on top of the safing. The previously unfastened end of the  
10 composite is then fastened to the floor slab. Intumescent caulk is used to seal the composite to the safing and insulation, as described herein.

The three layer flexible composite materials of the invention, and systems employing them, remedy a long felt need to both thermally insulate and provide fire barrier properties for dynamic wall/floor joints, joints which  
15 heretofore have provided chimneys for growing fires in multi-level buildings. The other flexible composite materials and systems of the invention provide new means to fireproof construction gaps.

Other advantages of the invention will become apparent from the detailed description which follows.

20                                    Brief Description of the Drawing

FIG. 1 shows a side elevation view of a prior art thermally insulated dynamic wall/floor joint;

FIGs. 2 and 3 show cross-sectional views of two embodiments of flexible composite materials of the invention useful as fire barriers in dynamic  
25 and static joints, with layers exaggerated in thickness;

FIGs. 4-6 show cross-sectional views of three embodiments of flexible composite materials of the invention useful as fire barriers in static joints or in "short" systems for dynamic joints; and

FIGs. 7-9 show side elevation views of three "long" embodiments of  
30 systems of the invention useful as fire barriers for dynamic joints.

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Description of Preferred Embodiments

In order to meet the shortcomings in building standards for dynamic joints noted by Nicholas, above, I have invented the flexible composites of FIGs. 2 and 3.

5 Thermal insulation (as shown in FIG. 1 at 12) is required by building codes for its thermal insulation and moisture barrier properties only. There are currently no codes or standards for fire barrier construction for dynamic joints because there has heretofore been no method to test dynamic joints for fire safety.

10 All flexible composite embodiments of the invention (except embodiment 40 of FIG. 4) utilize a flexible intumescent fire retardant composite layer or layers. The flexible intumescent fire retardant composite preferably comprises from 15 to 80 weight percent hydrated alkali metal silicate, from 15 to 40 weight percent of an organic binder, at most 40 weight percent of an organic  
15 char-forming component, and at most 50 weight percent filler. The organic binder may be formed from a binder precursor which is either thermally or radiation curable, or mixture of thermally and radiation curable binder precursors. Examples of typical and preferred intumescent fire retardant composites are disclosed in U.S. Pat. No. 4,273,879. This composition is a  
20 flexible rubbery material in its unexpanded state, but once subjected to temperatures on the order of 110°C and higher, intumesces up to 10 times its original volume and becomes a rigid char which is capable of sealing penetrations in which it is contained against the passage of smoke, vapors, and water. Other intumescent fire retardant materials may be used, such as those  
25 known under the trade designations "Palusol" (BASF) and "Expantrol" (3M).

The inorganic fiber layer preferably comprises up to 98 percent by weight of inorganic fibers, and from 2 to 20 weight percent organic and/or inorganic binder. Useful inorganic fibrous materials include alumina-silicate fibers commercially available under the trade designations "Cerafiber" from  
30 Manville Corporation, "Kaowool" from Thermal Ceramics, and "Fiberfrax" from Carborundum Company, soft glass fibers commercially available under

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the trade designation "E-glass" from Manville Corporation, mineral wool, and refractory filaments such as zirconia-silica fibers and crystalline alumina whiskers. Suitable organic binders include rubber latices such as natural rubber, styrene-butadiene, butadiene-acrylonitrile, acrylates, and methacrylates.

- 5 Suitable inorganic binders include bentonite and colloidal silica. Small amounts of surfactants, foaming agents, and flocculating agents may also be used if necessary.

A particularly preferred inorganic fiber layer useful in flexible composites of the invention is a mixture of 50 weight percent mineral wool and  
10 50 weight percent alumina-silicate fibers, bound together at points of mutual contact by an acrylic latex binder. An example of a method of making such an inorganic fiber layer is presented as Example 1.

FIGs. 2 and 3 illustrate cross-sectional views of the flexible composites of the invention useful in systems of the invention for both dynamic and static  
15 joints. FIG. 2 shows a flexible composite 20 which includes a first layer 28 of inorganic fibers and binder, a second layer 26 of metal foil adhered to the first layer by a suitable adhesive, and a third layer 29 of a flexible intumescent fire retardant composite. Third layer 29 is partially intermingled between individual fibers of inorganic fiber layer 28 during a coating process (described below)  
20 and requires no adhesive to bond to the first layer.

The adhesive used to bond the metal foil layer 26 to the inorganic fiber layer 28 may be a pressure-sensitive adhesive or a thermoplastic material. For ease of processing, the metal foil and adhesive are preferably in the form of an aluminum foil/acrylic pressure-sensitive adhesive tape, such as that known  
25 under the trade designation "T-49", from 3M. Preferred pressure-sensitive adhesives, because of their extended shelf life and resistance to detackifying under atmospheric conditions, are the acrylic-based copolymer adhesives, such as those disclosed in U.S. Pat. No. Re 24,906.

It may be preferable to use thermoplastic or thermosetting polymeric  
30 adhesives, for example, in environments where an acrylic-based adhesive might prematurely crack. Thermoplastic polymers useful and preferred as adhesives

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include polyethylene and polypropylene. Thermosetting polymeric adhesives which are useful and preferred include the reaction product of a diisocyanate (such as toluene diisocyanate {TDI} and the like) and a polyester. One example of this latter adhesive found useful in the present invention is available  
5 from Morton Chemical Company, Chicago, IL, under the trade designation "Adcote".

Embodiment 30 of FIG. 3 is similar in all respects to embodiment 20 of FIG. 2, except that the first or middle layer 32 includes unexpanded vermiculite. Unexpanded vermiculite will expand to up to 10 times its original  
10 volume upon exposure to a temperature of 300°C. As noted previously, unexpanded vermiculite comprises no more than 65 weight percent of the total weight of layer 32.

FIG. 4 illustrates in cross-section a flexible composite embodiment suitable for use as a fire barrier for static joints. Illustrated is layer 28 of  
15 inorganic fibers and binder with a coating of metal foil 26 laminated thereto, as above described.

In embodiments 20, 30, and 40, the metal foil is preferably either aluminum or one of the stainless steels, such as type 304, and the like. The foil thickness may range from 10 micrometers to 200 micrometers, preferably  
20 from 30 to 70 micrometers. Thickness of metal foil may be increased to provide a stronger flexible composite and for lessening the chance of breaking the inorganic fiber/foil laminate in the process of coating the intumescent fire retardant coating thereon. However, metal foils having thickness above 200 micrometers significantly reduces the flexibility of the flexible composites, thus  
25 making the task of installation more difficult. Thicker foils are also not preferred from an economic standpoint.

FIGs. 5 and 6 illustrate, again in cross-sectional views, flexible composites 50 and 60 in accordance with the present invention. Flexible composites 50 and 60 are suitable for use as fire barriers for static and dynamic  
30 joints, but only "short" embodiments of the latter, i.e., those not covering the insulation. Flexible composite 50 includes layer 28 of inorganic fibers and

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binder, as in the previous embodiments, and two layers 29 of flexible intumescent fire retardant composite. Between the two layers 29 is a layer 34 of inorganic fabric, which lends strength to the flexible composite. Flexible composite 60, illustrated in cross-section in FIG. 6, includes a layer 28 of inorganic fiber and binder, as in all embodiments, sandwiched between two layers 34 of inorganic fabric. Composite 60 is completed by having two layers 29 of flexible intumescent fire retardant composite material as the external layers. As with flexible composite 50, the inorganic fabric layers 34 increase the strength of the flexible composite so that the flexible composites withstand the hose stream test of ASTM E-119.

The inorganic fabric in embodiments 50 and 60 preferably comprises glass fiber.

FIGs. 7-9 illustrate "long" systems of the invention. System embodiment 70 of FIG. 7 is one particularly preferred system of the invention. Either of the flexible composite embodiments 20 or 30 of FIGs. 2 and 3 may be used with this system to pass the time-temperature test utilized in ASTM E-119 and E-814 tests, or their equivalent (test described in Example 6). FIG. 7 shows essentially the same features as FIG. 1 with the addition of the flexible composite, denoted as having two portions 72 and 74. First portion 72 extends from mullion attachment pin 17 horizontally and then vertically past attachment pin 18, between the insulation 12 and safing 14. Second portion 74 of the flexible composite includes the "S" shaped portion which is accumulated over the upper surface of safing 14, being attached to floor 20 by attachment pin 19. It will of course be understood by those of skill in the art that a plurality of attachment pins 17, 18, and 19 may be required, and that configurations other than "S-shaped" accumulations of flexible composite may be employed.

Installation of the system shown in FIG. 7 may be either as a retrofit, wherein the thermal insulation 12 and safing 14 have been previously installed, for example, in an older building. Alternatively, the insulation, safing, and fire barrier may be installed as new construction. In either case, installation is simple and cost effective. The flexible composite 72 is first attached to the



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mullion 13 via pin 17. If the safing is already present, one must remove at least a portion of the safing to "thread" flexible composite 72 up to the next floor of the building. Thereafter safing 14 is inserted, and flexible composite portion 74 is accumulated over the safing, care being taken that the intumescent fire retardant side faces the interior of the building. It should be apparent to those skilled in the art that the terms "first" and "second" portions of the flexible composite do not mean that the flexible composite is necessarily in two separate pieces. As this embodiment illustrates, portions 72 and 74 are actually portions of a single flexible composite sheet.

Intumescent caulk 75 is placed where shown in FIG. 7 to provide additional fume and smoke barriers. Preferably, a flexible intumescent fire retardant caulk is used having composition similar to the intumescent fire retardant composite described previously. Caulks known under the trade designations "CP 25WB", "CP 25 N/S", "CP 25S/L", and "Firedam", available from 3M, are particularly well suited for this purpose.

After caulk is in place the unattached end of flexible composite 74 is attached to the floor 20 via pin(s) 19.

The system shown in FIG. 8 as embodiment 80 of the invention is an alternative to the embodiment of FIG. 7. In embodiment 80, safing 14 extends from the butt end of the floor to the exterior glass, with thermal insulation 12 positioned above and below safing 14. In this system, all thermal insulation 12 can be glass fiber as long as safing 14 is mineral wool or similar high temperature resistant material. A first portion of flexible composite 72 of either FIGs. 2 or 3 is installed parallel to the exterior glass of the building, intumescent side facing toward the exterior glass. A second portion 74 is attached to the first portion with caulk, as previously described, and accumulated over the upper surface of safing 14 and attached as illustrated to floor 20. In this embodiment, safing 14 is thus partially supported by Z-clip(s) 77 and by the lower thermal insulation panel 12.

As a fire develops in the vicinity of safing 14, underneath floor slab 20, if thermal insulation 12 is glass-fiber it will eventually disintegrate as heat

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builds. However, the flexible intumescent layer of the first portion of flexible composite 72 will intumesce and fill the gap between the exterior glass and safing, thus ensuring that no fire, smoke, or fumes enter the space above floor 20. This is true even if the wall and floor deform since slack exists in flexible composite portion 74.

FIG. 9 shows another embodiment wherein the "long" flexible composite is used, again with either flexible composite embodiment of FIGs. 2 and 3. As with the embodiment shown in FIG. 7, thermal insulation 12 and safing 14 may in this case be either glass fiber or high temperature resistant mineral wool or the like. Flexible composite portion 72, beginning at attachment pin 16, is positioned adjacent and parallel to thermal insulation 12, with metal foil facing the interior of the building. Portion 72 is installed so that it hangs down vertically until it reaches a point near the safing, where it is formed a "U" shape, and attached via pin 19 to floor 20. Note that this construction allows the intumescent material to face downward, toward the fire in the vicinity of the safing. A second portion of flexible composite 74 is accumulated over the upper surface of safing 14, as in FIGs. 7 and 8. For added support for safing 14, a Z-clip is preferably included in the construction as illustrated.

Preferably the solvent used in the method of making the intumescent precursor solution is water or an organic solvent, the organic solvent selected from the group consisting of lower alkyl ketones (e.g. methyl ethyl ketone and the like), aromatic hydrocarbons (such as benzene, xylene, and the like), and other hydrocarbon solvents.

The method of making the laminate of step (i) is not within the scope of this invention, although an example is presented below which teaches how to make one preferred inorganic fiber/metal foil laminate useful in the flexible composites of the invention. Briefly, the inorganic fiber mat of the laminate is made using conventional paper making techniques, and an adhesive used to adhere the metal layer to the inorganic fiber layer. The adhesive may either be a pressure-sensitive adhesive or a thermoplastic. The latter may be preferred to

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prolong the life of the flexible composites of the invention, as many pressure-sensitive adhesives tend to become brittle with the passage of time.

After the inorganic fiber/metal foil laminate is produced, an intumescent precursor solution is coated onto the laminate on the side of the laminate not  
5 having foil. Coating of the intumescent precursor solution may be performed by any of a number of ways within the scope of the invention. Preferred methods use horizontal or vertical (tower) coating machines.

The preferred method of curing the intumescent fire retardant composite is by the application of heat, although the use of radiation curable organic  
10 binders alone or in combination with thermally curable binders may be preferred in certain circumstances.

When thermally curable binders are used to form the intumescent fire retardant composite, conventional roll, flow bar, and knife coating machines may be utilized to apply the intumescent precursor solution (comprising the  
15 intumescent fire retardant material and a solvent) to an exposed major surface of the inorganic fiber layer.

The number of drying ovens, temperature of the ovens, number of coats of precursor solution applied, and thickness of the individual coats may vary.

The method of the invention may be further understood with reference to  
20 the following examples, in which all parts and percentages are by weight unless otherwise stated.

### Examples

Example 1: Production of an Inorganic Layer Comprising  
25 Mineral Wool and Alumina-silicate Fibers/  
Aluminum Foil Laminate

The component ingredients and amounts used to make the preferred inorganic fiber layer/metal foil laminate are presented in Table 1. The mineral wool was first slushed in a pulper, charging one half of the total weight of  
30 mineral wool (300 kg) and 12.7 kiloliters (kl) of water. This charge was slushed for 15 seconds, and subsequently pumped to a cyclone cleaner (known under the trade designation "Krebs"). The flow through the cyclone cleaner was adjusted to 1 kl/min at 25 psig pressure. The two effluents ("accept"

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effluent and "reject" effluent) from the cyclone cleaner were inspected for shot and percent solids after the flow had stabilized through the accept effluent conduit using a 2.54 cm diameter orifice. The reject rate was 5-10 % of the weight of original mineral wool charge. The accept effluent was subsequently  
 5 fed into a precipitation chest.

The above steps were repeated with the remaining mineral wool, with a proportionate amount of water.

After two batches of mineral wool reached the precipitation chest and tested for percent solids, all of the alumina-silicate fibers were fed to the pulper  
 10 with 18.2 kl of water. The alumina-silicate fibers were slushed for 60 seconds in the pulper, and then pumped into the precipitation chest to form a slurry with the mineral wool.

**Table 1**

	<u>Ingredient</u>	<u>Amount (kg or ml)</u>
15	water	47.3
	mineral wool (Bethlehem)	604
20	alumina-silicate fibers ("Cerafiber", Manville Corporation)	613
25	sodium aluminate ("2372", Nalco Chemical)	20.4
	46% solids acrylic latex ("Rhoplex HA-8", Rohm & Haas)	409
30	defoaming agent ("Foammaster DF-160-LP")	0.91 ml
35	liquid alum	50.9 ml

The slurry of mineral wool fibers and alumina-silicate fibers was  
 40 precipitated by first adding sodium aluminate with mixing until the pH of the

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slurry reached 8.0 or above. The acrylic latex, defoamer, and liquid alum were then added in order, with the pH of the slurry after addition of the liquid alum checked to be sure it was 6.5 or below. The slurry was then pumped to a machine chest.

- 5        The inorganic fiber layer was produced by a conventional wet lay paper technique, dried, and wound on a reel without a liner. After the basis weight of a wet 10.2 cm x 15.2 cm sample of the web reached a target weight of 20-25 gm, a flexible composite of the invention was made by forcing aluminum foil tape (known under the trade designation "T-49", having aluminum
- 10 thickness of 50 micrometers and a polyester/TDI adhesive into the nip at the reel, with the adhesive of the tape facing the top side of the inorganic fiber layer.

- 15        Example 2:    Production of an Inorganic Layer Comprising 100% Alumina-silicate Fibers/Aluminum Foil Laminate

A second laminate was formed in exactly the same manner as in Example 1, the only difference being the deletion of mineral wool.

- 20        Example 3:    Production of Flexible Composite Using Example 1 Inorganic Layer and Water-based Intumescent Precursor Solution

- A length of 45.7 meters of 1.22 meter wide laminate of Example 1 was coated with an intumescent precursor solution comprising the ingredients listed
- 25 in Table 2. The intumescent precursor solution was 76% solids, and had a viscosity of 24,000 centipoise, measured using a Brookfield viscometer, Model "RV", #4 spindle, 6 rpm, at 20°C. This intumescent precursor solution was coatable and sprayable. The intumescent precursor solution was applied using a horizontal roll coater having three heating zones, with the first, second, and
- 30 third, heating zones having temperatures of 120°C, 143°C, and 154°C, respectively. The intumescent precursor solution was applied having a dry coating weight of 2260 gm/m<sup>2</sup> (gsm). This coating was blistered.

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**Table 2**

<u>Ingredients</u>	<u>Amount(parts)</u>
polychloroprene latex	8619
5 antifoam agent ("Foamaster")	49
water	49
10 surfactant ("Triton X-100")	203
surfactant ("Tamol 850")	203
agerite	101
15 zinc oxide	254
iron oxide (FeO)	254
zinc borate/sodium silicate	4325
20 ("Expantrol 4B")	
aluminum hydroxide	1524
25 glass fibers	509

**Example 4: Production of Flexible Composite Using Example 2 Inorganic Layer and Water-based Intumescent Precursor Solution**

30 A length of 36.6 meters of 1.22 meter wide laminate of Example 2 was coated using intumescent precursor solution made in accordance with Example 3. The intumescent precursor solution was again applied with a horizontal roll coater, with the same heating zone temperatures as used in Example 3. This coating was also blistered.

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**Example 5: Production of Flexible Composite Using Example 2 Inorganic Layer and Organic Solvent-based Intumescent Precursor Solution**

40 A length of 265 meters of 1.22 meter wide laminate of Example 2 was coated with an intumescent precursor solution comprising the ingredients listed in Table 3.

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**Table 3**

	<b><u>Ingredient</u></b>	<b><u>Amount(parts)</u></b>
5	xylene	22.5
	methyl ethyl ketone	22.5
10	calcium carbonate	16.6
	polychloroprene latex	15.7
	sodium silicate	9.3
15	zinc borate	1.8
	chlorinated olefins	4.5
20	2-ethylhexyldiphenyl phosphate	2.8
	water	2.6

25

The intumescent precursor solution had a viscosity of 40,000 centipoise, measured using a Brookfield viscometer model "RV", #4 spindle, 6 rpm, at 20°C. A horizontal roll coater was used, as in Examples 3 and 4. Using first, second, and third heating zone temperatures of 93°C, 99°C, and 107°C, respectively, and machine speed of 3 meters/min, the coating did not cure. The heating zone temperatures were then raised to 121°C, 143°C, and 154°C, which produced cured coatings having coating weight (dry) of 1033 gsm. Blistered coatings were produced.

35 **Example 6:** Production of Flexible Composite Using Example 2 Inorganic Layer (Containing Vermiculite), and Organic Solvent-based Intumescent Precursor Solution

As earlier disclosed, the inorganic fibrous layer used in the flexible composites of the invention may include vermiculite. This Example used a commercial mat available under the trade designation "Interam 2100", commercially available from 3M, which had aluminum foil adhesive tape

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(known under the trade designation "T-49") laminated thereto. This inorganic fibrous layer was essentially the same as that of Example 2, except the layer included 50 weight percent cationically exchanged vermiculite, as disclosed in U.S. Pat. No. 4,305,992, which is incorporated by reference. The inorganic  
5 fibrous layer of this Example was coated with an intumescent precursor solution made in accordance with Example 5.

#### Comparative Example A

For comparison in the fire rating and hose stream tests (described  
10 below), a commercially available aluminum foil/mineral wool laminate (known under the trade designation "Thermafiber Life Safety System", from United States Gypsum Company) was employed as Comparative Example A.

In making the flexible composites of the invention in the above Examples 1-6, a horizontal roll coater was employed. A tower knife coater may also be  
15 employed. It has been found that in the case of a tower knife coater (wherein the inorganic fiber/metal foil laminate is threaded and pulled vertically through two serially arranged vertical heating zones, with one intumescent precursor solution coated onto the laminate in each zone), the first vertical heating zone temperature should generally be 90°C while the second vertical heating zone  
20 temperature is preferably 110°C. These conditions are appropriate for coating an intumescent caulk known under the trade designation "CP 25N/S" (3M) diluted to 55% solids with methyl ethyl ketone, at wet thickness of 0.2 cm, pulling the laminate through the tower coater at a rate of 55 meters/hr. Spray coating has also been evaluated for the water-based  
25 intumescent precursor solutions with some success.

In some of the flexible composites made by the above procedures (both in horizontal and tower coaters), blistering of the flexible intumescent coatings during heating occurred. This was believed due to the entrapment of solvent in bubbles as the composite passed through the heating zones of the dryers.  
30 Through suitable adjustment of speed through the zones, and/or lowering of the coating thickness, blistering was overcome. It was also noted that the location



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where the precursor slurry was pumped into the coating pan was where blistering seemed to occur most. The occurrence of blisters was substantially eliminated by judicious placement of the feed to the coating pan.

Another problem was splice failures. High tension was needed to pull the coated laminates through the ovens or heating zones. Splices made with high temperature adhesive on film backing, with long (60-90 cm) longitudinal strips on each side of the splice worked well; however, normal, short splices would fail midway through the heating zones, especially in vertical tower coaters. Even with a splice failure, however, splices could be remade in the oven without a significant loss of product. Starting and stopping the horizontal and tower coating processes was also performed quickly. The most effective splice tape found for this coating process was a 20.3 cm wide pressure-sensitive tape having a high temperature polyester/TDI thermoset adhesive.

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#### Test Method

A test procedure was developed by the assignee of this invention so that systems comprising thermal insulation and the flexible composites of the invention could be evaluated in dynamic joints. The test was developed to simulate a "real world" fire in which the vertical wall and floor deform. Test "specimens" were constructed as shown in FIG. 7. Floor slab 20 was concrete and had thickness of 11.43 cm, while in place of spandrel glass 10, a 10.2 cm thick, vertical concrete slab was used. Two pieces of 5.1 cm X 2.54 cm aluminum mullion were installed vertically, separated by 61 cm, with a 2.54 cm gap between the vertical concrete slab and mineral wool insulation, also of 10.2 cm thickness. The safing was 10.2 cm thick mineral wool. The flexible composite to be tested was installed as shown in FIG. 7, and the structure heated from below using time-temperature curves of ASTM tests E-119 and E-814. A thermocouple was placed near the "cold" side of the safing (near the thermal insulation) and the temperature recorded at 20 minute intervals. The results for Examples 2, 5, 6, and Comparative Example A are presented in Table 1.

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Hose stream tests in accordance with ASTM E-119 and E-814 were also performed on the inventive Examples and Comparative Example A. The water spray was 6.1 meters from center, using 45 seconds sweep time and at 0.2 megaPascals water pressure. The results for the hose stream test (denoted "HS") are presented in Table 4 as "P" for "pass" and "F" for "fail".

**Table 4: Results of Fire Test and Hose Stream Test**  
Temperature (°C) at Time (min.)

Example	20	40	60	80	120	140	160	180	H S
2	27	63	88	91	93	96	107	121	P
5	26	49	66	71	82	96	99	107	P
6	29	46	63	71	82	116	124	135	P
A	38	68	96	88	182	204	210	227	F

Whether a fire barrier passes the fire test is determined by noting the cold side temperature after 1 hour (ASTM E-814) and 2 hours (ASTM E-119). The cold side temperature should be below 163°C + ambient temperature to pass ASTM E-814, and below 121°C + ambient temperature to pass ASTM E-119. As may be seen by the data in Table 4, even the aluminum foil/inorganic fibrous laminate of Example 2 would pass the fire rating test and hose stream test; however, since intumescent layer is present in Example 2, there would still be a chimney for smoke and possibly toxic fumes. Note that the Comparative Example did not pass either the equivalent of the ASTM E-119 time-temperature test or the hose stream test.

Various modifications and alterations of this invention will become apparent to those skilled in the art without departing from the scope and spirit of this invention, and it should be understood that this invention is not to be unduly limited to the illustrative embodiments set forth herein.

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What is claimed is:

1. A flexible composite material, suitable for use as a fire barrier for static joints, characterized by having:

(a) a first layer material having first and second major surfaces, the first  
5 layer material being inorganic fibers and a binder in the form of a flexible mat;  
and

(b) a layer of metal foil adhered to the first layer by an adhesive, the metal foil having a melting temperature of at least 350°C.

10 2. Flexible composite material in accordance with claim 1 further characterized by having a third layer (c) adhered to the second major surface of the first layer material, the third layer material being an intumescent fire retardant composite material.

15 3. Flexible composite material in accordance with claim 1 further characterized by said inorganic fibers being selected from the group consisting of alumina-silicate fibers, mineral wool fibers, glass fibers, refractory filaments, crystalline alumina whiskers, and mixtures thereof.

20 4. Flexible composite material in accordance with claim 1 further characterized by said metal foil being selected from the group consisting of aluminum and stainless steel.

25 5. Flexible composite material in accordance with claim 2 further characterized by said intumescent fire retardant material being from 15 to 80 weight percent hydrated alkali metal silicate, from 15 to 40 weight percent of an organic binder, at most 40 weight percent of an organic char-forming component, and at most 50 weight percent filler.

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6. A flexible composite material, suitable for use as a fire barrier, characterized by having:

(a) a first layer material having first and second major surfaces, the first layer material being inorganic fibers and a binder in the form of a flexible mat;

5 (b) a second layer material adhered to the first major surface of the first layer material, the second layer material being an intumescent fire retardant composite material;

(c) a third layer adhered to the second layer, the third layer being inorganic fibers; and

10 (d) a fourth layer adhered to the third layer, the third layer being an intumescent fire retardant composite material.

7. A flexible composite material, suitable for use as a fire barrier, characterized by having:

15 (a) a first layer material having first and second major surfaces, the first layer material being inorganic fibers and a binder in the form of a flexible mat;

(b) second and third layers adhered to the first and second major surfaces of the first layer, respectively, the second and third layers being  
20 inorganic fibers;

(c) fourth and fifth layers adhered to the second and third layers, respectively, the fourth and fifth layers being an intumescent fire retardant composite material.

25 8. A system for thermally insulating and fire-proofing an exterior wall and an exterior wall/floor joint of a building, wherein the wall includes a mullion and exterior sheet material, and wherein the floor includes a material which is rigid at room temperatures but experiences sagging due to its weight at fire temperatures, the system characterized by having:

30 (a) an insulating component positioned within the shape defined by the mullion upon attachment to the mullion and having interior and exterior facing

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surfaces, the insulating component being an inorganic material capable of providing thermal insulation for the building;

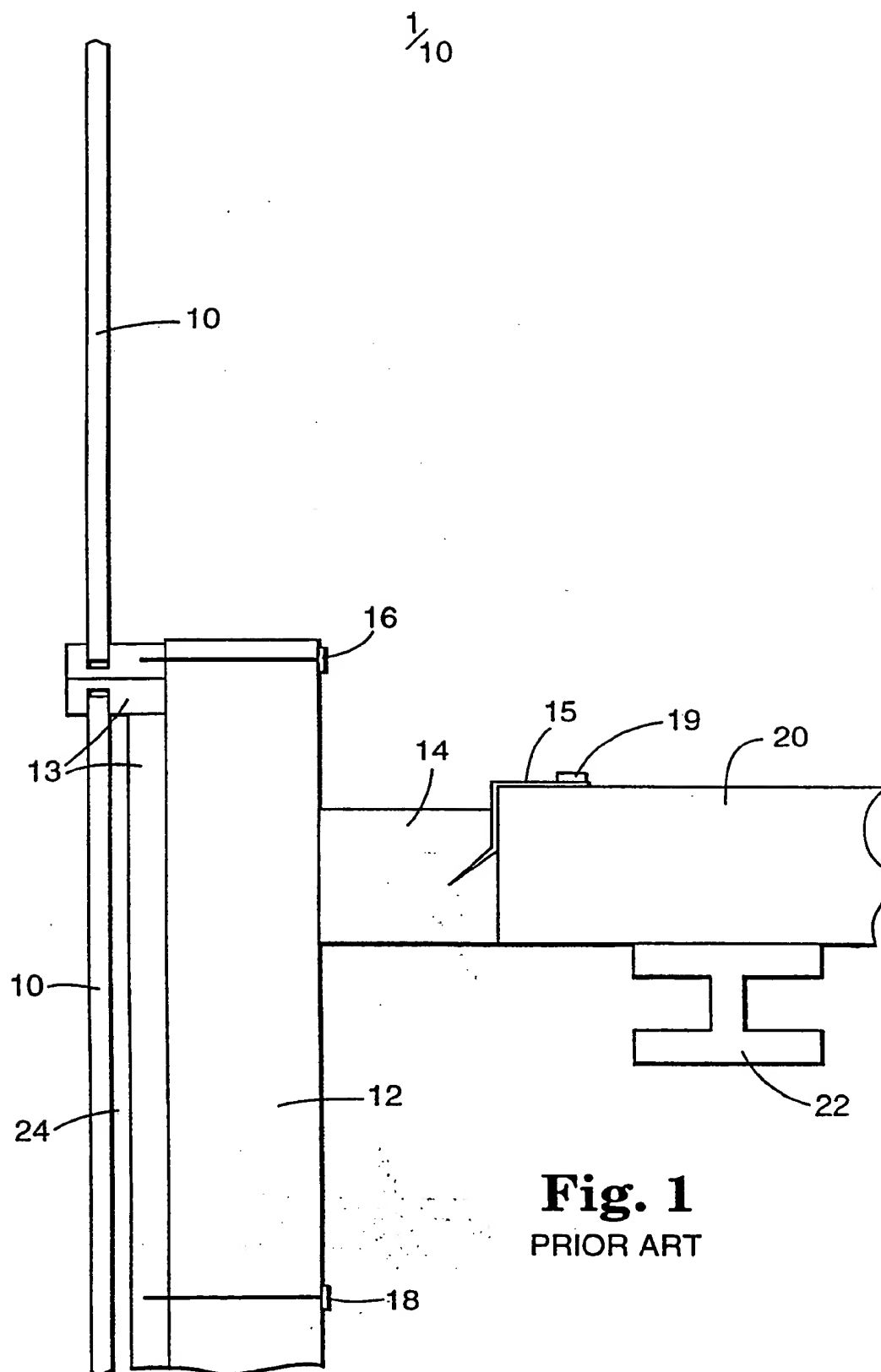
(b) a safing component positioned between an exterior butt end of the floor and insulating component; and

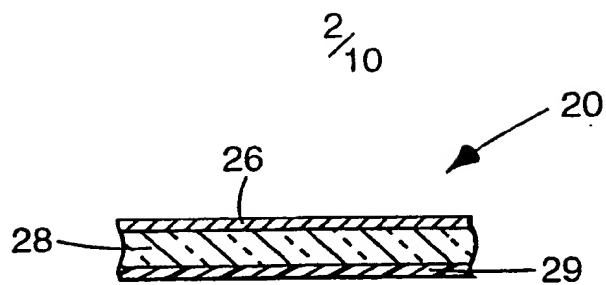
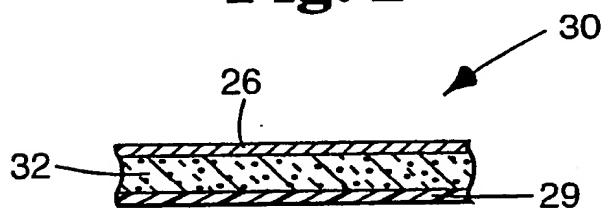
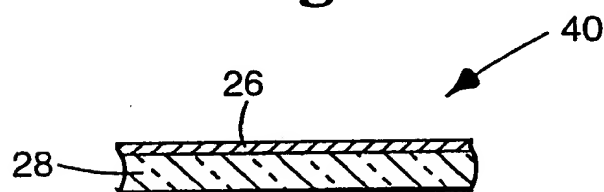
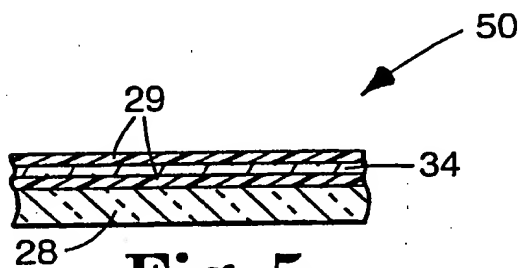
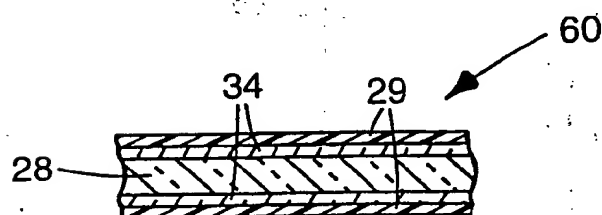
- 5       (c) a fire barrier flexible composite material, the fire barrier having first and second components, the first component positioned adjacent and parallel to the insulating component, and the second component positioned above the safing component in a curved fashion, the second component having first and second ends, the first end attached to the top surface of the floor and the second  
10 end attached to the first component, and the first component of the fire barrier attached to the mullion, wherein the second component has at least one curved portion which provides slack, thus allowing the fire barrier to remain intact during relative movement of the wall and floor.

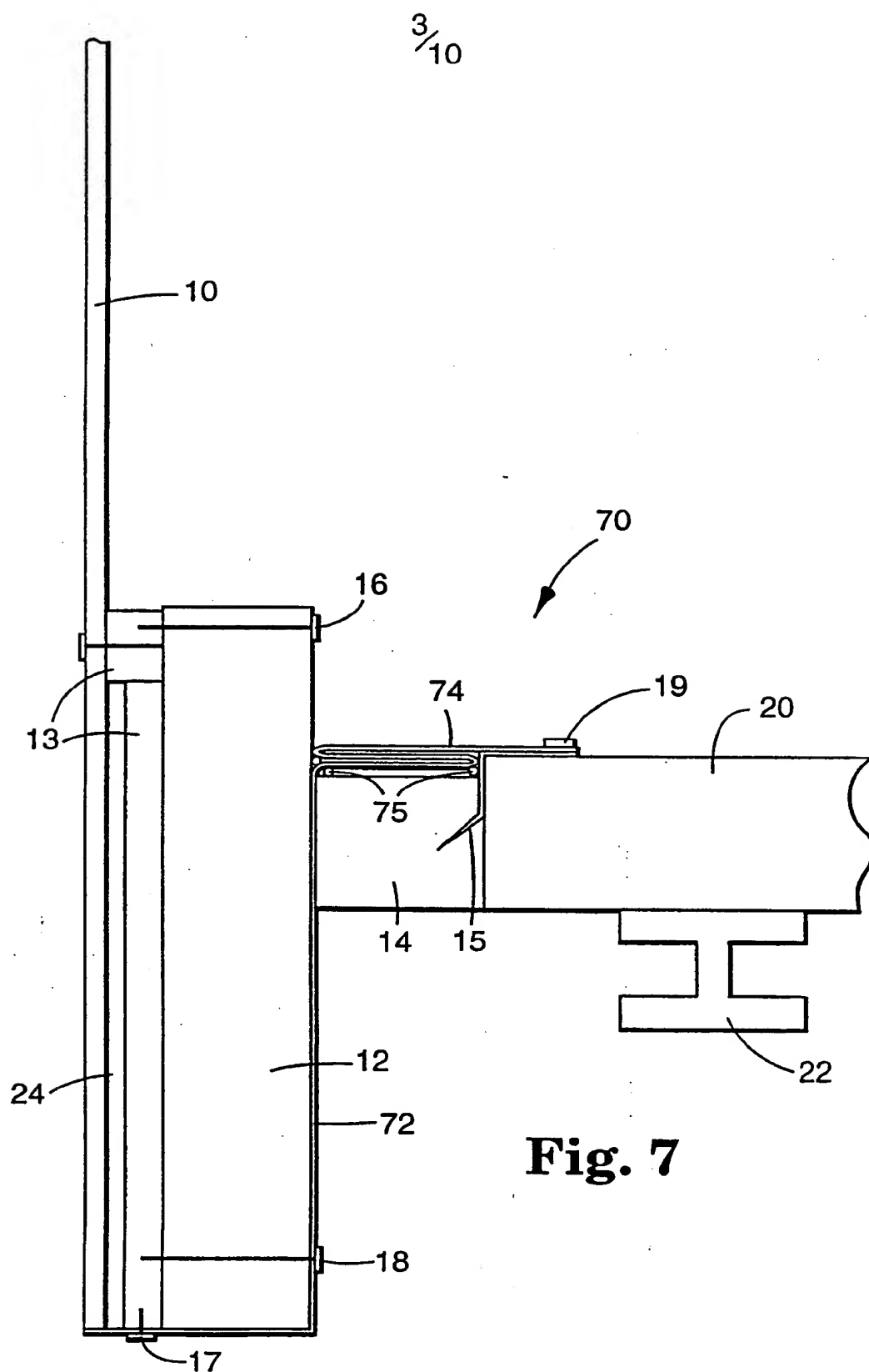
- 15       9. The system of claim 8 further characterized by the first component of the fire barrier being positioned adjacent the interior facing surface of the insulating component and having a lower terminus attached to the mullion and an upper terminus attached to the second fire barrier component, the second terminus extending between the insulating component and safing component, the  
20 second component being an S-shaped curve, and wherein the safing is supported by one or more Z-clips.

10. The system of claim 9 further characterized by the flexible composite material being:

- 25       (a) a first layer material having first and second major surfaces, the first layer material being inorganic fibers and a binder in the form of a flexible mat;  
      (b) a second layer material of metal foil adhered to the first layer by an adhesive, the metal foil having a melting point of at least 350°C; and  
      (c) a third layer adhered to the second major surface of the first layer  
30 material, the third layer material being an intumescent fire retardant composite material.

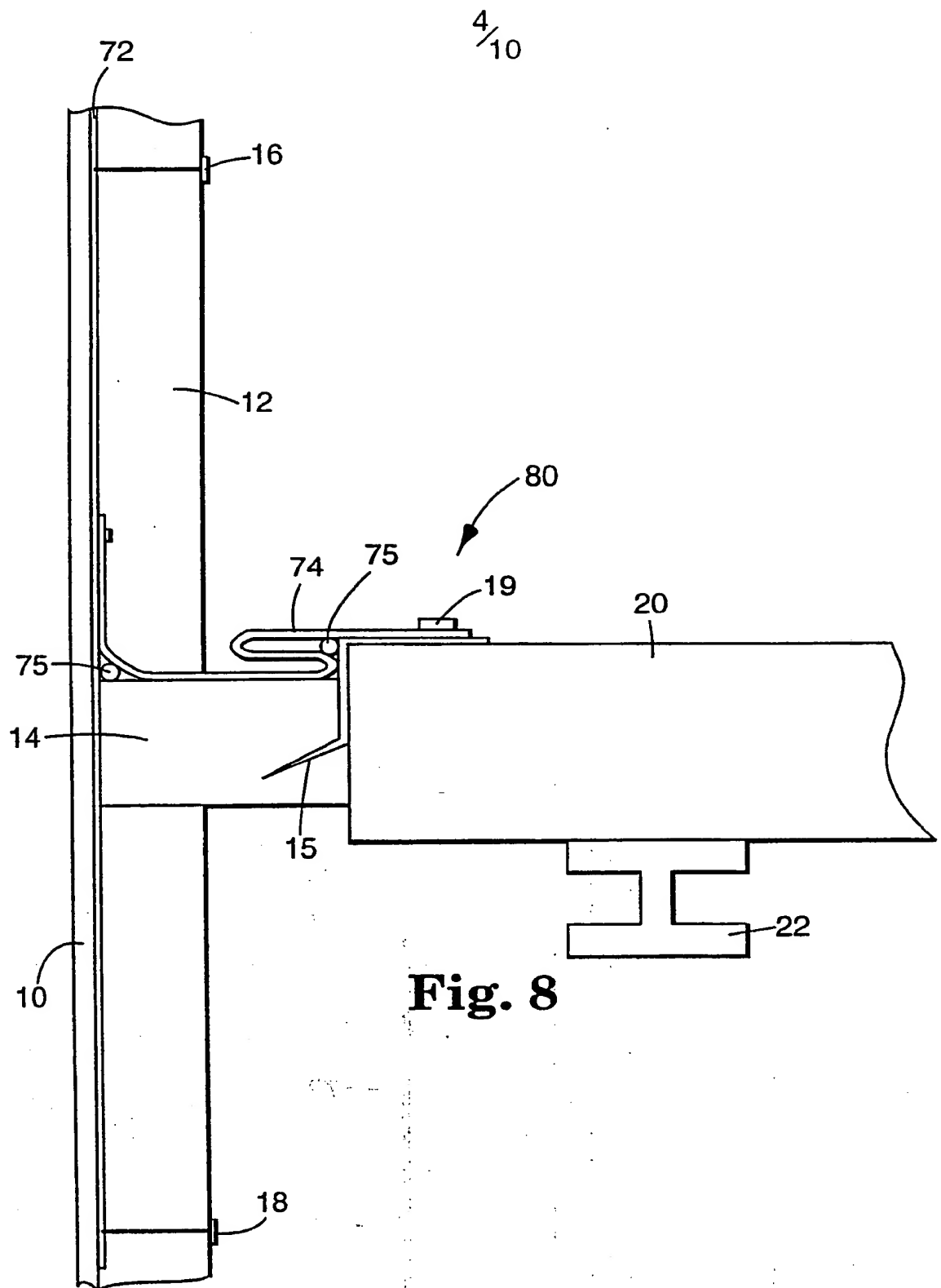


**Fig. 2****Fig. 3****Fig. 4****Fig. 5****Fig. 6**

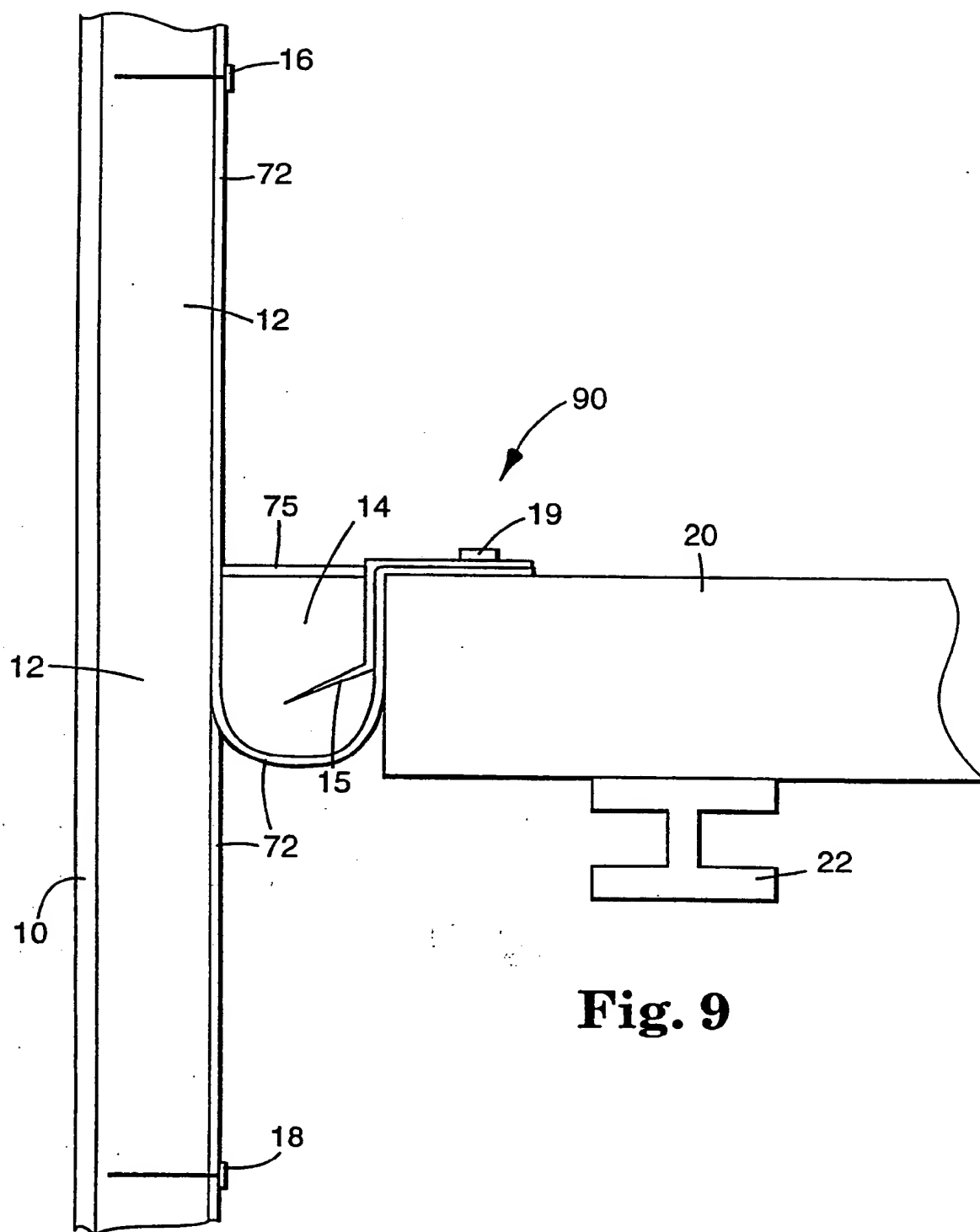


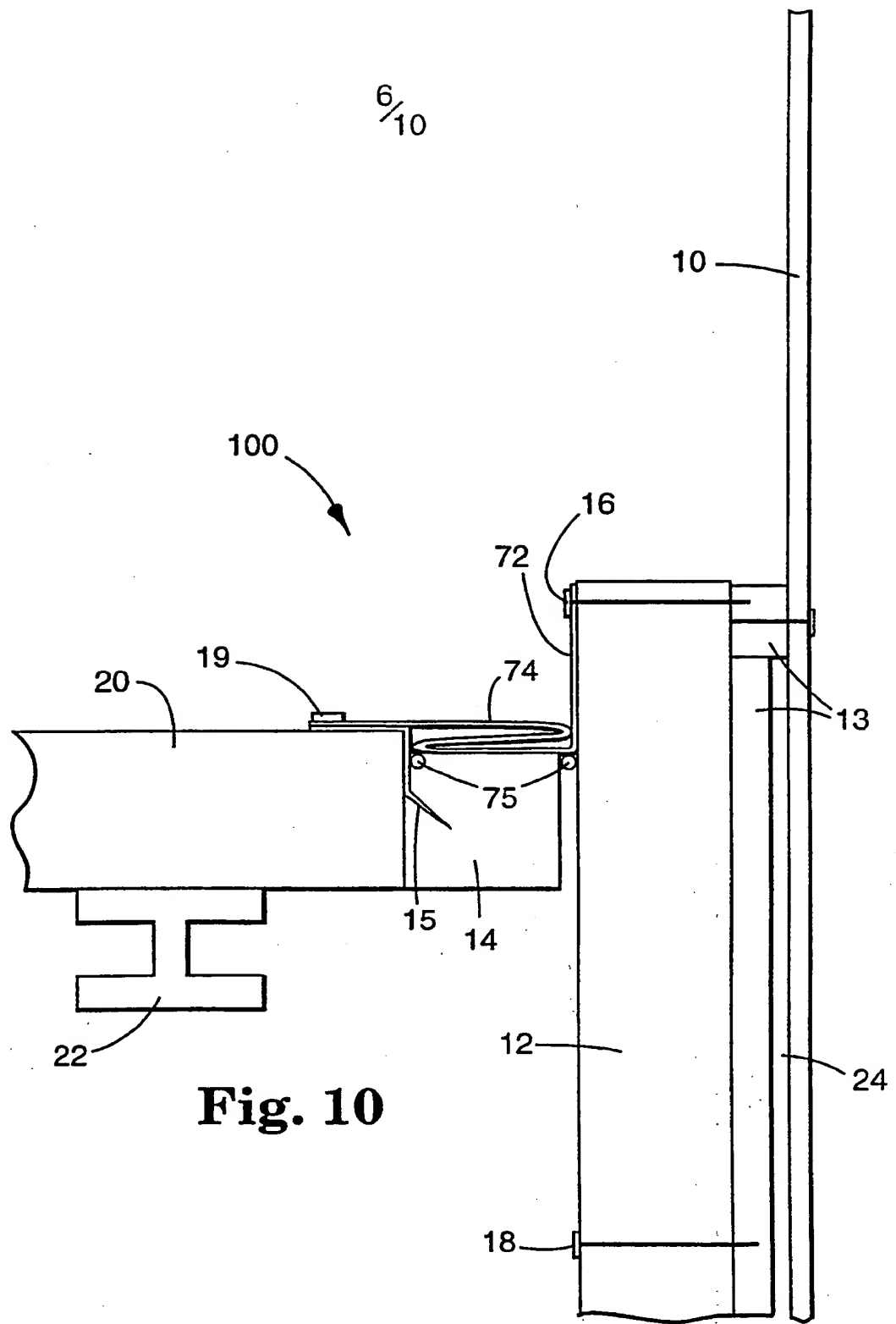
**Fig. 7**

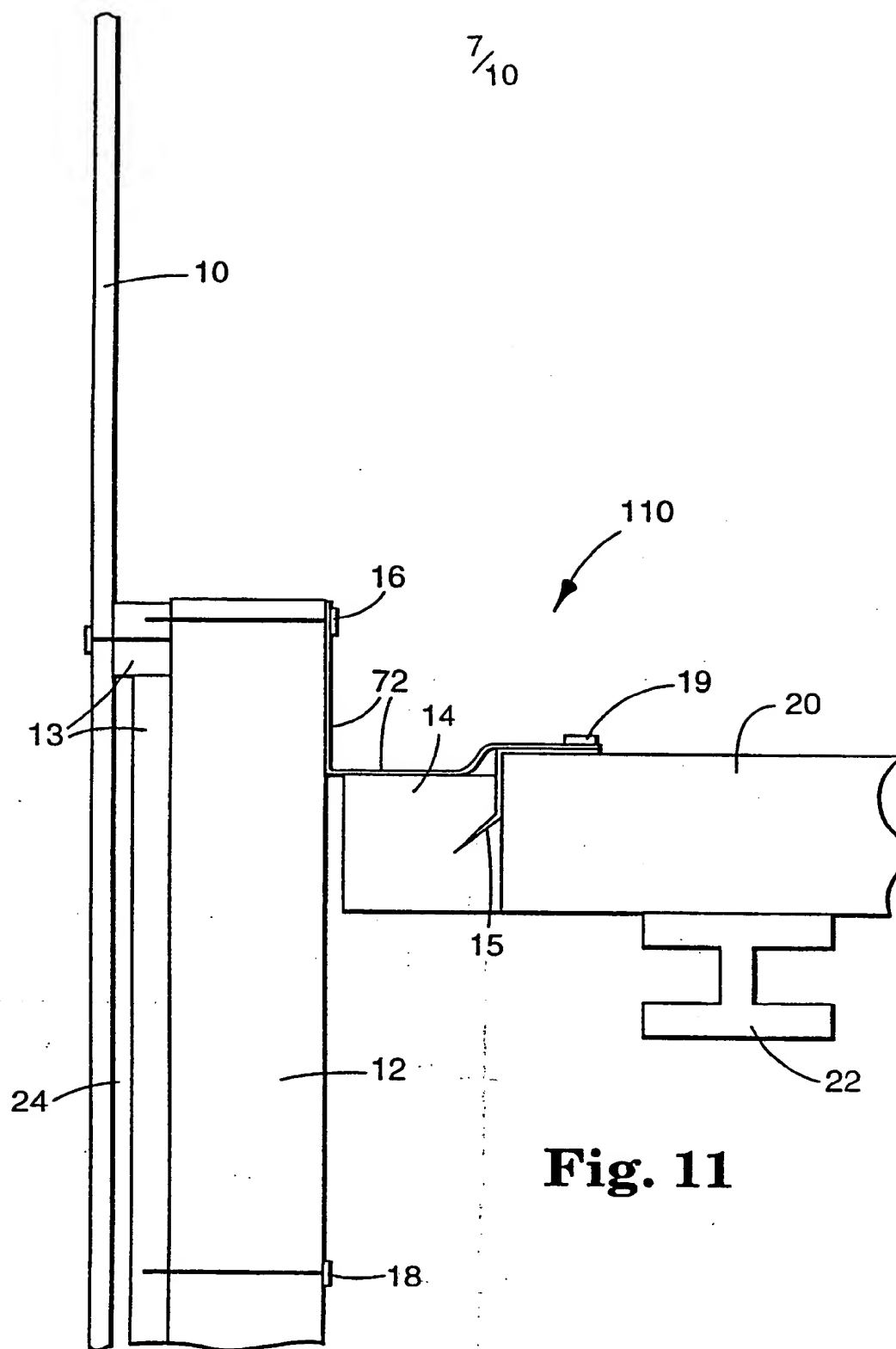




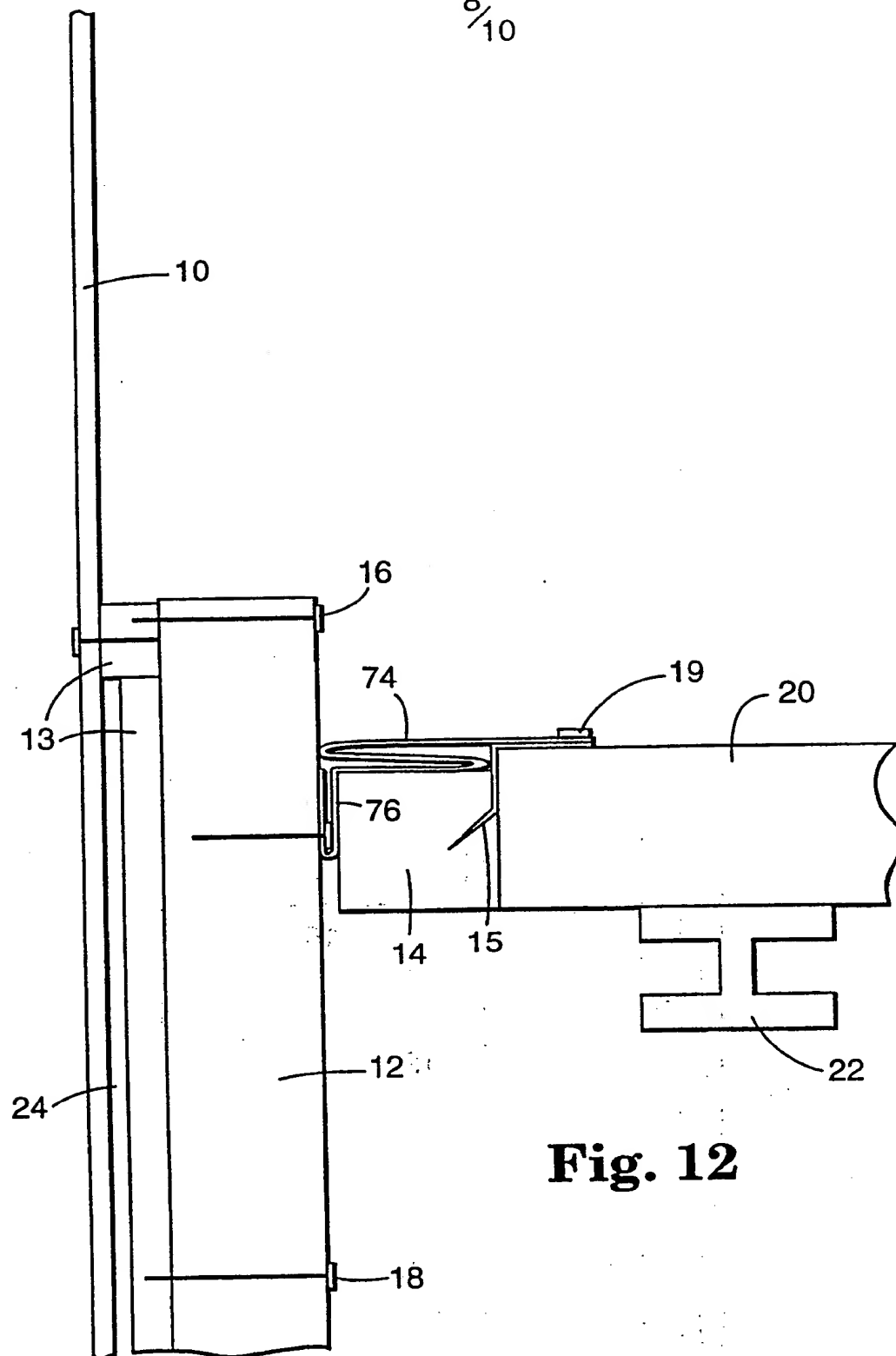
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**Fig. 9**



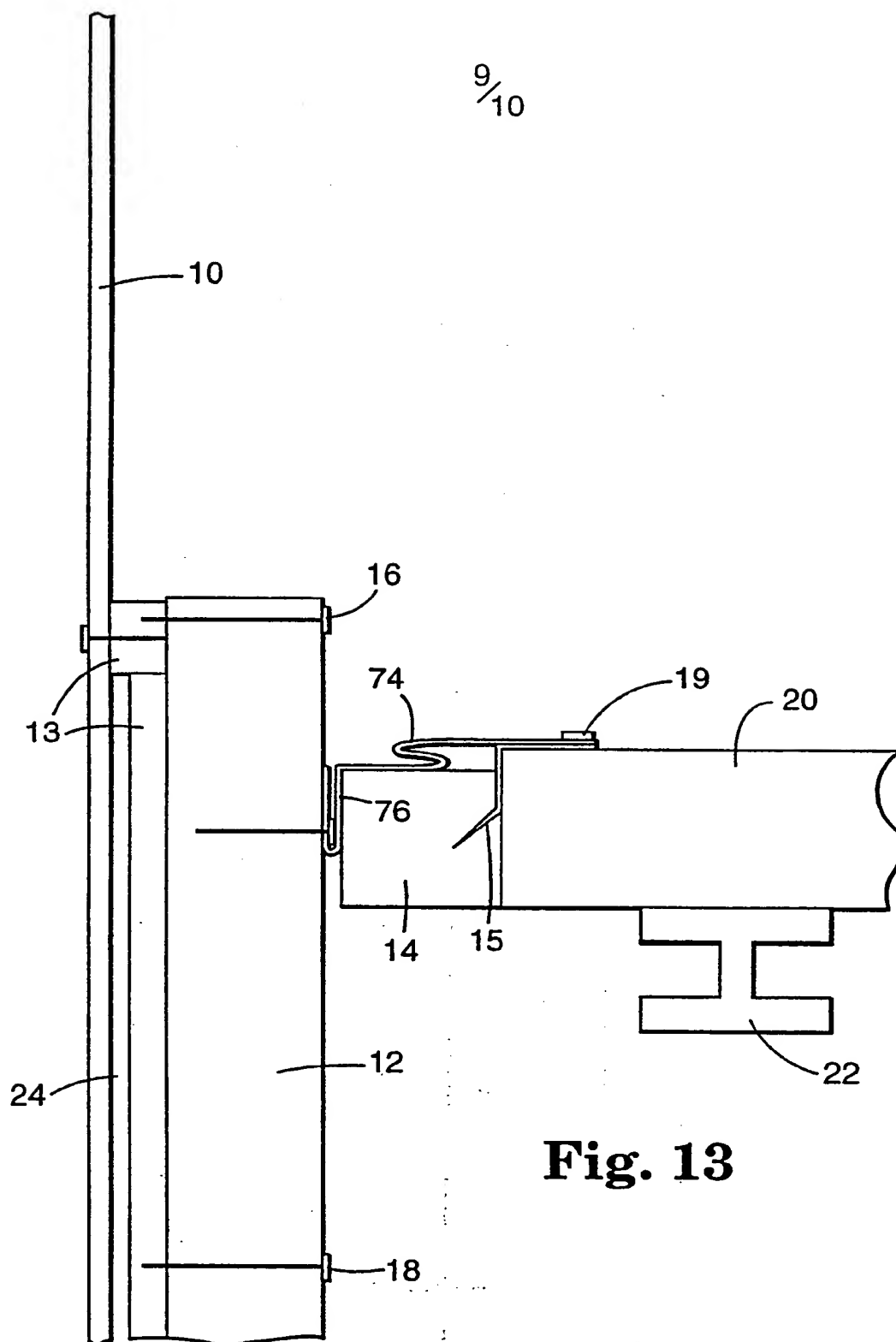
**Fig. 11**

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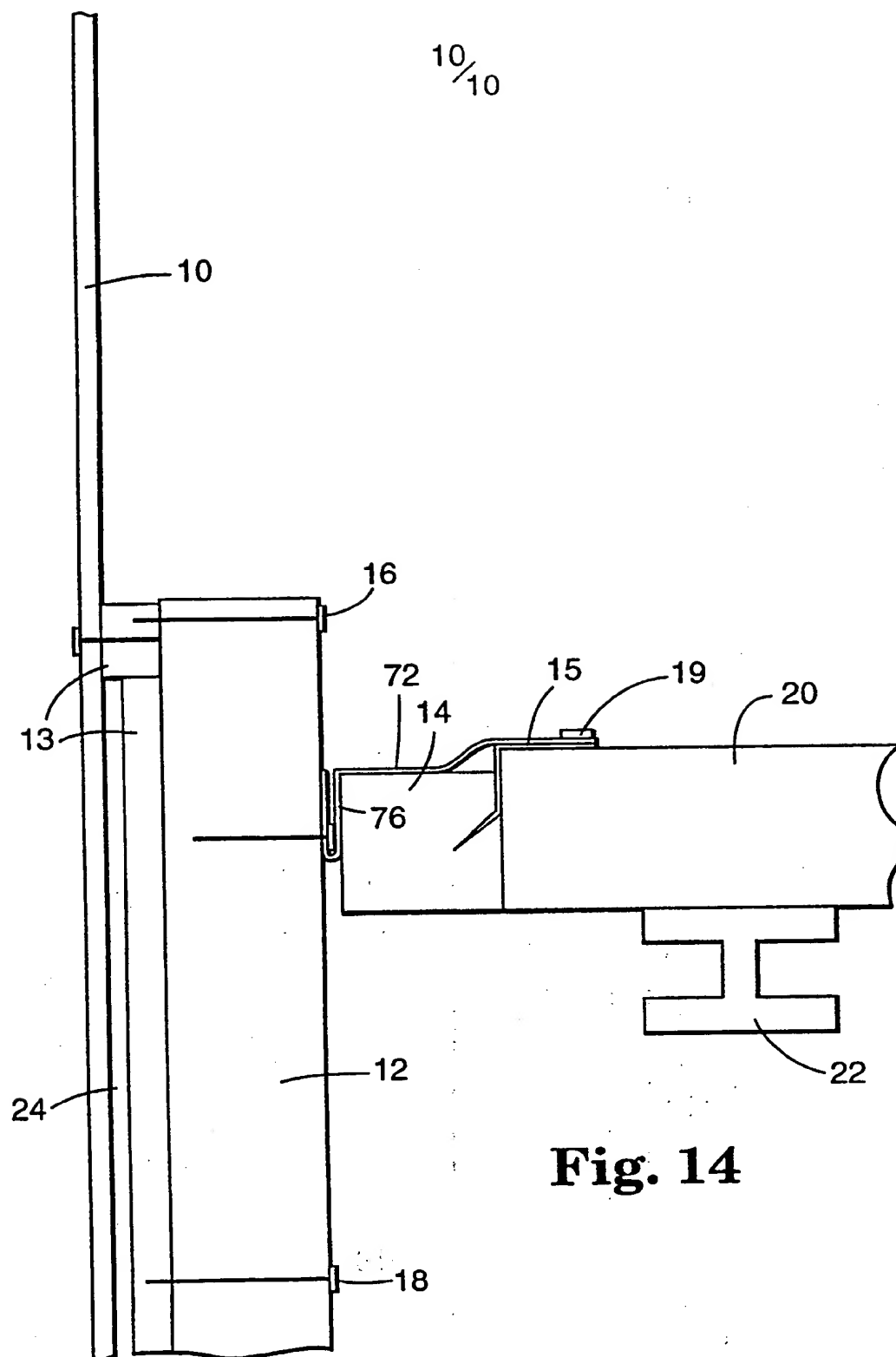


**Fig. 12**

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**Fig. 13**

**Fig. 14**

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 93/03551

<b>I. CLASSIFICATION OF SUBJECT MATTER</b> (if several classification symbols apply, indicate all) <sup>1</sup> According to International Patent Classification (IPC) or to both National Classification and IPC IPC <sup>5</sup> : B 32 B 7/00, E 04 B 1/94		
<b>II. FIELDS SEARCHED</b>		
Classification System	Minimum Documentation Searched <sup>2</sup>	
Classification Symbols IPC <sup>5</sup> : B 32 B 7/00, B 32 B 5/00, B 32 B 15/00, B 32 B 17/00, B 32 B 19/00, E 04 B 1/00		
Documentation Searched other than Minimum Documentation to the extent that such Documents are included in the Fields Searched <sup>3</sup>		
<b>III. DOCUMENTS CONSIDERED TO BE RELEVANT</b> <sup>4</sup>		
Category <sup>5</sup>	Citation of Document, <sup>6</sup> with indication, where appropriate, of the relevant passages <sup>12</sup>	Relevant to Claim No. <sup>13</sup>
A	US, A, 4 256 799 (OHASHI) 17 March 1981 (17.03.81), column 3, lines 31-40. --	1
A	WO, A1, 89/09 316 (AGFK) 05 October 1989 (05.10.89), fig. 1a, 3; page 3, lines 12-16. --	1
P, A	GB, A, 2 250 933 (ENVIRONMENTAL SEALS) 24 June 1992 (24.06.92), examples 2, 4; page 2, lines 21-27. --	2, 6, 7
A	US, A, 4 977 719 (LA ROCHE et al.) 18 December 1990 (18.12.90), fig. 11; column 6, lines 38-64; column 2, lines 43-44 --	1, 8
<div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p><sup>14</sup> Special categories of cited documents: <sup>15</sup></p> <p><sup>16</sup> "A" document defining the general state of the art which is not considered to be of particular relevance</p> <p><sup>17</sup> "E" earlier document but published on or after the international filing date</p> <p><sup>18</sup> "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p><sup>19</sup> "O" document referring to an oral disclosure, use, exhibition or other means</p> <p><sup>20</sup> "P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p><sup>21</sup> "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p><sup>22</sup> "X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p><sup>23</sup> "Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p><sup>24</sup> "Z" document member of the same patent family</p> </div> </div>		
<b>IV. CERTIFICATION</b>		
Date of the Actual Completion of the International Search <div style="text-align: center;">29 June 1993</div>		Date of Mailing of this International Search Report <div style="text-align: center;">18.08.93</div>
International Searching Authority <div style="text-align: center;">EUROPEAN PATENT OFFICE</div>		Signature of Authorized Officer <div style="text-align: center;">ONDER e.h.</div>

Form PCT/ISA/210 (second sheet) (January 1985)



III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, " with indication, where appropriate, of the relevant passages	Relevant to Claim No.
	(cited in the application).	

## ANHANG

## ANNEX

## ANNEXE

zum internationalen Recherchen-  
bericht über die internationale  
Patentanmeldung Nr.

to the International Search  
Report to the International Patent  
Application No.

au rapport de recherche inter-  
national relatif à la demande de brevet  
international n°

PCT/US 93/03551 SAE 73586

In diesem Anhang sind die Mitglieder  
der Patentfamilien der im obenge-  
nannten internationalen Recherchenbericht  
angeführten Patentdokumente angegeben.  
Diese Angaben dienen nur zur Unter-  
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This Annex lists the patent family  
members relating to the patent documents  
cited in the above-mentioned inter-  
national search report. The Office is  
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La présente annexe indique les  
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relatifs aux documents de brevets cités  
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national visée ci-dessus. Les renseigne-  
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In Recherchenbericht angeführtes Patentdokument Patent document cited in search report Document de brevet cité dans le rapport de recherche	Datum der Veröffentlichung Publication date Date de publication	Mitglied(er) der Patentfamilie Patent family member(s) Membre(s) de la famille de brevets	Datum der Veröffentlichung Publication date Date de publication
US A 4256799	17-03-81	GB A1 2030516 GB B2 2030516 JP A2 55027268 JP B4 58052492 JP A2 55027267 JP B4 58052491 US A 4292361	10-04-80 01-12-82 27-02-80 22-11-83 27-02-80 22-11-83 29-09-81
WD A1 8909316	05-10-89	keine - none - rien	
GB A 2250933		EP A1 492977 GB A0 9027786 GB A1 2250933	01-07-92 13-02-91 24-06-92
US A 4977719	18-12-90	AU A1 36072/89 EP A1 347865 JP A2 2043433 US A 4866898	21-12-89 27-12-89 14-02-90 19-09-89